3.0 QUALITY CONTROL/QUALITY ASSURANCE OPERATIONS- PLM

A program to routinely assess the quality of the results produced by the PLM laboratory must be developed and implemented. **Quality Control (QC)** is a system of activities whose purpose is to control the quality of the product or service so that it meets the need of the users. This also includes **Quality Assessment**, whose purpose is to provide assurance that the overall quality control is being done effectively. While the essential elements of a quality control system are described in detail elsewhere;[1,2,3,4,5,6] only several of the elements will be discussed here. **Quality Assurance (QA)** is comprised of Quality Control and Quality Assessment and is a system of activities designed to provide assurance that a product or service meets defined standards of quality.

The purpose of the Quality Assurance program is to minimize failures in the analysis of materials prior to submitting the results to the client. Failures in the analysis of asbestos materials include false positives, false negatives, and misidentification of asbestos types. False positives result from identification or quantitation errors. False negatives result from identification, detection, or quantitation errors.

For the stereomicroscopic and PLM techniques, the quality control procedures should characterize the accuracy and precision of both individual analysts and the techniques. Analysts should demonstrate their abilities on calibration materials, and also be checked routinely on the analysis of unknowns by comparison with results of a second analyst. The limitations of the stereomicroscopic and PLM techniques can be determined by using a second analytical technique, such as gravimetry, XRD, or AEM. For example, stereomicroscopic and PLM techniques can fail in the analysis of floor tiles because the asbestos fibers in the sample may be too small to be resolved by light microscopy. An XRD or AEM analysis is not subject to the same limitations, and may indicate the presence of asbestos in the sample.

The accuracy, precision, and detection limits of all analytical techniques described in this method are dependent on the type of sample (matrix components, texture, etc.), on the preparation of the sample (homogeneity, grain size, etc.), and the specifics of the method (number of point counts for PLM, mass of sample for gravimetry, counting time for XRD,

etc.). These should be kept in mind when designing quality control procedures and characterizing performance, and are variables that must be tracked in the quality assurance system.

3.1 General Considerations

3.1.1 Training

Of paramount importance in the successful use of this or any other analytical method is the well-trained analyst. It is highly recommended that the analyst have completed course work in optical mineralogy on the collegiate level. That is not to say that others cannot successfully use this method, but the classification error rate[7] may, in some cases, be directly attributable to level of training. In addition to completed course work in optical mineralogy, specialized course work in PLM and asbestos identification by PLM is desirable. Experience is as important as education. A good laboratory training program can be used in place of course work. Analysts that are in training and not yet fully qualified should have all analyses checked by a qualified analyst before results are released. A QC Plan for asbestos identification would be considered incomplete without a detailed description of the analyst training program, together with detailed records of training for each analyst.

3.1.2 Instrument Calibration and Maintenance

Microscope alignment checks (alignment of the polarizer at 90° with respect to the analyzer, and coincident with the cross-lines, proper orientation of the slow vibration direction of the Red I compensator plate, image of the field diaphragm focussed in the plane of the specimen, centering of the central dispersion staining stop, etc.) should be performed with sufficient frequency to ensure proper operations. Liquids used for refractive index determination and those optionally used for dispersion staining should have periodic refractive index checks using a refractometer or known refractive index solids. These calibrations must be documented.

Microscopes and ancillary equipment should be maintained daily. It is recommended that at least once per year each microscope be thoroughly cleaned and re-aligned by a professional microscope service technician. Adequate inventories of replaceable parts

(illumination lamps, etc.) should be established and maintained. All maintenance must be documented.

3.2 Quality Control of Asbestos Analysis

3.2.1 Qualitative Analysis

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite) using combined stereomicroscopic and PLM techniques. Standards for the six asbestos types listed are available from NIST, and should be used to train analysts in the measurement of optical properties and identification of asbestos. These materials can also be used as identification standards for XRD and AEM.

Identification errors between asbestos types (e.g. reporting amosite when tremolite is present) implies that the analyst cannot properly determine optical properties and is relying on morphology as the identification criteria. This is not acceptable. Each analyst in the lab should prove his or her proficiency in identifying the asbestos types; this can be checked through use of calibration materials (NVLAP proficiency testing materials, materials characterized by an independent technique, and synthesized materials) and by comparing results with another analyst. The identification of all parameters (e.g. refractive indices, birefringence, sign of elongation, etc.) leading to the identification should fall within control limits determined by the laboratory. In addition, a subset of materials should be analyzed using another technique to confirm the analysis.

As discussed earlier, the qualitative analysis is dependent upon matrix and asbestos type and texture. Therefore, the quality assurance system should monitor for samples that are difficult to analyze and develop additional or special steps to ensure accurate characterization of these materials. When an analyst is found to be out of the control limits defined by the laboratory, he or she should undergo additional training and have confirmatory analyses performed on all samples until the problem has been corrected.

3.2.2 Quantitative Analysis

The determination of the amount of asbestos in a sample can be accomplished using the various techniques outlined in this method. The mandatory stereomicroscopic and PLM examinations provide concentrations in terms of volume, area, or weight, depending upon the calibration procedure. Gravimetric and quantitative XRD techniques result in concentrations in units of weight percent. Specific guidelines for determining accuracy and precision using these techniques are provided in the appropriate sections of this method. In general, however, the accuracy of any technique is determined through analysis of calibration materials which are characterized by multiple independent techniques in order to provide an unbiased value for the analyte (asbestos) in question. The precision of any technique is determined by multiple analyses of the sample. The analyst is the detector for stereomicroscopic and PLM techniques, as opposed to gravimetric and XRD techniques, and therefore must be calibrated as an integral part of the procedure.

As in the qualitative analysis, the laboratory should determine its accuracy and precision for quantitative asbestos analysis according to the type of material analyzed and the technique used for analysis. For example, the laboratory may determine that its analysts have a problem with calibrated area estimates of samples containing cellulose and chrysotile and therefore needs to make or find special calibration materials for this class of sample.

Calibration materials for quantitative analysis of asbestos are available through the Bulk Asbestos NVLAP as proficiency testing materials for those laboratories enrolled in NVLAP. In a report provided following a test round, the concentration of asbestos in each sample is given in weight percent with 95%/95% tolerance limits, along with a description of the major matrix components. Materials from other round robin and quality assurance programs for asbestos analysis may not have been analyzed by independent techniques; the concentrations may represent consensus PLM results that could be significantly biased. Therefore, values from these programs should <u>not</u> be used as calibration materials for quantitative analysis.

Calibration materials for quantitative analysis can also be synthesized by mixing asbestos and appropriate matrix materials, as described in Appendix C of this method. These

materials are usually simplifications of "real world" samples; therefore the accuracy and precision determined from analysis of these materials are probably ideal.

Limits on permissible analytical variability must be established by the laboratory prior to QC implementation. It is recommended that a laboratory initially be at 100% quality control (all samples reanalyzed.) The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%. Quantitative results for standards including the mean and error estimate (typically 95% confidence or tolerance intervals) should be recorded. Over time these data can be used to help determine control limits for quality control charts.

The establishment and use of control charts is extensively discussed elsewhere in the literature. [1,2,3,4,5] Several cautions are in order:

- Control charts are based on the assumption that the data are distributed normally. Using rational subgrouping, the means of the subgroups are approximately normally distributed, irrespective of the distribution of the individual values in the subgroups. Control charts for asbestos analysis are probably going to be based on individual measurements, not rational subgroups. Check the data for normality before proceeding with the use of control charts. Ryan[8] suggests a minimum of 50 analyses before an attempt is made to establish control limits. However, for this analysis, consider setting "temporary" limits after accumulating 20-30 analyses of the sample.

- Include both prepared slides as well as bulk samples in your reference inventory.

- Make certain that sample quantities are sufficient to last, and that the act of sampling will not alter the composition of the reference sample.

Data on analytical variability can be obtained by having analysts repeat their analyses of samples and also by having different analysts analyze the same samples.

3.3 Interlaboratory Quality Control

The establishment and maintenance of an interlaboratory QC program is fundamental to continued assurance that the data produced within the laboratory are of consistent high quality. Intralaboratory programs may not be as sensitive to accuracy and precision error, especially if the control charts (see Section 3.2.2) for all analysts in the laboratory indicate small percent differences. A routine interlaboratory testing program will assist in the detection of internal bias and analyses may be performed more frequently than proficiency

testing. Arrangements should be made with at least two (preferably more) other laboratories that conduct asbestos identification by PLM. Samples (the number of which is left to the participating laboratories, but at least 4-10) representing the types of samples and matrices routinely submitted to the lab for analysis should be exchanged with sufficient frequency to determine intralaboratory bias. Both reference slides and bulk samples should be used. Results of the interlaboratory testing program should be evaluated by each of the participating laboratories and corrective actions, if needed, identified and implemented. Since quantitation problems are more pronounced at low concentrations ($\leq$ 5%), it would be prudent to include approximately 30-50% from this concentration range in the sample selection process.

3.4 Performance Audits

Performance audits are independent quantitative assessments of laboratory performance. These audits are similar to the interlaboratory QC programs established between several laboratories, but with a much larger cohort (the EPA Asbestos Bulk Sample Analysis Quality Assurance Program had as many as 1100 participating laboratories). Participation in this type of program permitted assessment of performance through the use of "consensus" test materials, and served to assist in assessing the bias relative to individual interlaboratory, as well as intralaboratory programs. Caution should be exercised in the use of "consensus" quantitation results, as they are likely to be significantly responsible for the propagation of high bias in visual estimates. The current NIST/NVLAP[9] for bulk asbestos laboratories (PLM) does not use concensus quantitation results. Results are reported in weight percent with a 95% tolerance interval. The American Industrial Hygiene Association (AIHA)[10] also conducts a proficiency testing program for bulk asbestos laboratories. Quantitation results for this program are derived from analyses by two reference laboratories and PLM, XRD and gravimetric analysis performed by Research Triangle Institute.

3.5 Systems Audits

Where performance audits are quantitative in nature, systems audits are qualitative. Systems audits are assessments of the laboratory quality system as specified in the Laboratory

Quality Assurance Manual. Such an audit might consist of an evaluation of some facet of the QA Manual, or the audit may be larger in scope. For example, the auditor might request specific laboratory data sheets which will be evaluated against written procedures for data recording in the laboratory. Or, the auditor might request air monitoring or contamination control data to review for frequency of sampling, analysis methodology, and/or corrective actions taken when problems were discovered. The audit report should reflect the nature of the audit as well as the audit results. Any recommendations for improvement should also be reflected in such a report.

3.6 References

1. **Quality Assurance for Air Pollution Measurement Systems. Volume I, Principles.** EPA-600/9-76-005, March, 1976.

2. Juran, J. and F. Gryna, **Quality Planning Analysis,** 2nd edition, McGraw-Hill, Inc., 1980.

3. Taylor, J.R., **Quality Control Systems,** McGraw Hill, Inc., 1989.

4. Ratliff, T.A., **The Laboratory Quality Assurance System,** Van Nostrand Reinhold, 1990.

5. Taylor, J.K., **Quality Assurance of Chemical Measurements,** Lewis Publishers, 1987.

6. Bulk Asbestos Handbook, National Institute of Standards and Technology, National Voluntary Laboratory Accreditation Program, NISTIR 88-3879, October 1988.

7. Harvey, B.W., "Classification and Identification Error Tendencies in Bulk Insulation Proficiency Testing Materials," **American Environmental Laboratory, 2**(2), 4/90, pp. 8-14.

8. Ryan, T.P., **Statistical Techniques for Quality Improvement,** John Wiley & Sons, Inc., New York, 1989.

9. National Institute of Standards & Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Building 411, Room A124, Gaithersburg, MD 20899, telephone (301) 975-4016.

10. American Industrial Hygiene Association (AIHA), 2700 Prosperity Avenue, Suite 250, Fairfax, VA 22031, (703) 849-8888.

APPENDIX A

Glossary Of Terms

APPENDIX A. GLOSSARY OF TERMS

Accuracy - The degree of agreement of a measured value with the true or expected value.

Anisotropic - Refers to substances that have more than one refractive index (e.g. are birefringent), such as nonisometric crystals, oriented polymers, or strained isotropic substances.

Asbestiform (morphology) - Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5μm. Aspect ratios should be determined for fibers, not bundles.
- Very thin fibrils, usually less than 0.5 micrometers in width, and
- Two or more of the following:
  - Parallel fibers occurring in bundles,
  - Fiber bundles displaying splayed ends,
  - Matted masses of individual fibers, and/or
  - Fibers showing curvature

These characteristics refer to the population of fibers as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of $<$20:1 for fibers longer than 5μm. If a sample contains a fibrous component of which most of the fibers have aspect ratios of $<$20:1 and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos.

Asbestos - A commercial term applied to the asbestiform varieties of six different minerals. The asbestos types are chrysotile (asbestiform serpentine), amosite (asbestiform grunerite), crocidolite (asbestiform riebeckite), and asbestiform anthophyllite, asbestiform tremolite, and asbestiform actinolite. The properties of asbestos that caused it to be widely used commercially are: 1) its ability to be separated into long, thin, flexible fibers; 2) high tensile strength; 3) low thermal and electrical conductivity; 4) high mechanical and chemical durability, and 5) high heat resistance.

Becke Line - A band of light seen at the periphery of a specimen when the refractive indices of the specimen and the mounting medium are different; it is used to determine refractive index.

Bias - A systematic error characterized by a consistent (non-random) measurement error.

Binder - With reference to a bulk sample, a component added for cohesiveness (e.g. plaster, cement, glue, etc.).

Birefringence - The numerical difference between the maximum and minimum refractive indices of an anisotropic substance. Birefringence may be estimated, using a Michel-Levy chart, from the interference colors observed under crossed polarizers. Interference colors are also dependent on the orientation and thickness of the grain, and therefore are used qualitatively to determine placement in one of the four categories listed below.

Qualitative	Quantitative(N-n)
none	0.00 or isotropic
low	≤0.010
moderate	0.011-0.050
high	>0.050

Bulk Sample - A sample of building material taken for identification and quantitation of asbestos. Bulk building materials may include a wide variety of friable and nonfriable materials.

Bundle - Asbestos structure consisting of several fibers having a common axis of elongation.

Calibration Materials - Materials, such as known weight % standards, that assist in the calibration of microscopists in terms of ability to quantitate the asbestos content of bulk materials.

Color - The color of a particle or fiber when observed in plane polarized light.

Compensator - A device with known, fixed or variable retardation and vibration direction used for determining the degree of retardation (hence the thickness or value of birefringence) in an anisotropic specimen. It is also used to determine the sign of elongation of elongated materials. The most common compensator is the first-order red plate (530-550nm retardation).

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Detection Limit - The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

Dispersion Staining (focal masking) - An optical means of imparting apparent or virtual color to transparent substances by the use of stops in the objective back focal plane; ir it is used to determine refractive indices.

Error - Difference between the true or expected value and the measured value of a quantity or parameter.

Extinction - The condition in which an anisotropic substance appears dark when observed between crossed polars. This occurs when the vibration directions in the specimen are parallel to the vibration directions in the polarizer and analyzer. Extinction may be complete or incomplete; common types include parallel, oblique, symmetrical and undulose.

Extinction Angle - For fibers, the angle between the extinction position and the position at which the fiber is parallel to the polarizer or analyzer privileged directions.

Fiber - With reference to asbestiform morphology, a structure consisting of one or more fibrils.

Fibril - The individual unit structure of fibers.

Friable - Refers to the cohesiveness of a bulk material, indicating that it may be crumbled or disaggregated by hand pressure.

Gravimetry - Any technique in which the concentration of a component is determined by weighing. As used in this document, it refers to measurement of asbestos-containing residues after sample treatment by ashing, dissolution, etc.

Homogeneous - Uniform in composition and distribution of all components of a material, such that multiple subsamples taken for analysis will contain the same components in approximately the same relative concentrations.

Heterogeneous - Lacking uniformity in composition and/or distribution of material; components not uniform. Does not satisfy the conditions stated for homogenous; e.g., layered or in clumps, very coarse grained, etc.

Isotropic - Refers to substances that have a single refractive index such as unstrained glass, un-oriented polymers and unstrained substances in the isometric crystal system.

Lamda Zero (λ_0) - The wavelength (λ_0) of the dispersion staining color shown by a specimen in a medium; both the specimen and medium have the same refractive index at that wavelength.

Matrix - Nonasbestos, nonbinder components of a bulk material. Includes such components as cellulose, fiberglass, mineral wool, mica, etc.

Michel-Levy Scale of Retardation colors - A chart plotting the relationship between birefringence, retardation and thickness of anisotropic substances. Any one of the three variables can be determined if the other two are known.

Morphology - The structure and shape of a particle. Characterization may be descriptive (platy, rod-like, acicular, etc) or in terms of dimensions such as length and diameter (see asbestiform).

Pleochroism - The change in color or hue of colored anisotropic substance when rotated relative to the vibration direction of plane polarized light.

Point Counting - A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used to determine the relative concentrations of asbestos minerals to nonasbestos sample components.

Polarization Colors - Interference colors displayed by anisotropic substances between two polarizers. Birefringence, thickness and orientation of the material affect the colors and their intensity.

Precision - The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the variability of results.

Reference Materials - Bulk materials, both asbestos-containing and nonasbestos-containing, for which the components are well-documented as to identification and quantitation.

Refractive Index (index of refraction) - The ratio of the velocity of light in a vacuum relative to the velocity of light in a medium. It is expressed as n and varies with wavelength and temperature.

Sign of Elongation - Referring to the location of the high and low refractive indices in an elongated anisotropic substance, a specimen is described as positive when the higher refractive index is lengthwise (length slow), and as negative when the lower refractive index is lengthwise (length fast).

Standard Reference Material (SRM) - A reference material certified and distributed by the National Institute of Standards and Technology.

Visual Estimate - An estimation of concentration of asbestos in a sample as compared to the other sample components. This may be a volume estimate made during stereomicroscopic examination and/or a projected area estimation made during microscopic (PLM) examination.

APPENDIX B

Apparatus For Sample Preparation And Analysis

B1.0 INTRODUCTION

The following lists the apparatus and materials required and suggested for the methods of sample preparation and analysis described in the test method.[1,2,3]

B2.0 STEREOMICROSCOPIC EXAMINATION

The following are suggested for routine stereomicroscopic examination.

- HEPA-filtered hood or class 1 biohazard hood, negative pressure
- Microscope: binocular microscope, preferably stereoscopic, 5-60X magnification (approximate)
- Light source: incandescent or fluorescent
- Tweezers, dissecting needles, scalpels, probes, etc. (for sample manipulation)
- Glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. (sample containers)

The following are suggested for sample preparation.

- Mortar and pestle, silica or porcelain-glazed
- Analytical balance (readability less than or equal to one milligram) (optional)
- Mill or blender (optional)

B3.0 POLARIZED LIGHT MICROSCOPY

The laboratory should be equipped with a polarized light microscope (preferably capable of Köhler or Köhler-type illumination if possible) and accessories as described below.

- Ocular(s) binocular or monocular with cross hair reticle, or functional equivalent, and a magnification of at least 8X
- 10X, 20X, and 40X objectives, (or similar magnification)

- Light source (with optional blue "day-light" filter)
- 360-degree rotatable stage
- Substage condenser with iris diaphragm
- Polarizer and analyzer which can be placed at 90 degrees to one another, and can be calibrated relative to the cross-line reticle in the ocular.
- Accessory slot for wave plates and compensators (or demonstrated equivalent).
- Wave retardation plate (Red I compensator) with approximately 550 nanometer retardation, and with known slow and fast vibration directions.
- Dispersion staining objective or a demonstrated equivalent. (optional)
- Monochromatic filter (n_D), or functional equivalent. (optional)

In addition, the following equipment, materials and reagents are required or recommended.[1]

- NIST traceable standards for the major asbestos types (NIST SRM 1866 and 1867)
- Class I biohazard hood or better (see "Note", Section 2.2.5)
- Sampling utensils (razor knives, forceps, probe needles, etc.)
- Microscope slides and cover slips
- Mechanical Stage
- Point Counting Stage (optional)
- Refractive index liquids: 1.490-1.570, 1.590-1.720 in increments of less than or equal to 0.005; high dispersion, (HD) liquids are optional; however, if using dispersion staining, HD liquids are recommended.
- Mortar and pestle
- Distilled water
- HCl, ACS reagent grade concentrated

- Muffle furnace (optional)
- Mill or blender (optional)
- Beakers and assorted glassware (optional)
- Other reagents (tetrahydrofuran, amyl acetate, acetone, sodium hexametaphosphate, etc.) (optional)

B4.0 GRAVIMETRY

The following equipment, materials, and reagents are suggested.

- Scalpels
- Crucibles, silica or porcelain-glazed, with lids
- Muffle furnace - temperature range at least to 500°C, temperature stable to $\pm$ 10°C, temperature at sample position calibrated to $\pm$ 10°C
- Filters, 0.4 μm pore size polycarbonate
- Petri dishes
- Glass filtration assembly, including vacuum flask, water aspirator, and/or air pump
- Analytical balance, readable to 0.001 gram
- Mortar and pestle, silica or porcelain-glazed
- Heat lamp or slide warmer
- Beakers and assorted glassware
- Centrifuge, bench-top
- Class I biohazard hood or better
- Bulb pipettes
- Distilled water
- HCl, reagent-grade concentrated

- Organic solvents (tetrahydrofuran, amyl acetate,etc)
- Ultrasonic bath

B5.0 X-RAY DIFFRACTION

Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- Mortar and pestle: agate or porcelain
- Razor blades
- Sample mill: SPEX, Inc., freezer mill or equivalent
- Bulk sample holders
- Silver membrane filters: 25-mm diameter, 0.45-μm pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006
- Microscope slides
- Vacuum filtration apparatus: Gelman No. 1107 or equivalent, the side-arm vacuum flask
- Microbalance
- Ultrasonic bath or probe: Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent
- Volumetric flasks: 1-L volume
- Assorted pipets
- Pipet bulb
- Nonserrated forceps
- Polyethylene wash bottle
- Pyrex beakers: 50-mL volume

- Desiccator
- Filter storage cassettes
- Magnetic stirring plate and bars
- Porcelain crucibles
- Muffle furnace or low temperature asher
- Class 1 biohazard hood or better

Sample Analysis

Sample analysis requirements include an x-ray diffraction unit, equipped with:

- Constant potential generator; voltage and mA stabilizers
- Automated diffractometer with step-scanning mode
- Copper target x-ray tube: high intensity; fine focus, preferably
- X-ray pulse height selector
- X-ray detector (with high voltage power supply): scintillation or proportional counter
- Focusing graphite crystal monochromator; or nickel filter (if copper source is used, and iron fluorescence is not a serious problem)
- Data output accessories:
  - Strip chart recorder
  - Decade scaler/timer
  - Digital printer

  or

  - PC, appropriate software and Laser Jet Printer
- Sample spinner (optional)
- Instrument calibration reference specimen: α-quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

Reagents, etc.

Reference Materials - The list of reference materials below is intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- Chrysotile: UICC Canadian, NIST SRM 1866 (UICC reference material available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK); (NIST Standard Reference Materials available from the National Institute of Standards and Technology, Office of Reference Standards, Gaithersburg, MD 20899)

- Crocidolite: UICC, NIST SRM 1866.

- "Amosite": UICC, NIST SRM 1866.

- Anthophyllite-Asbestos: UICC, NIST SRM 1867

- Tremolite Asbestos: Wards Natural Science Establishment, Rochester, NY; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 900064 (washed with dilute HCl to remove small amount of calcite impurity); Indian tremolite, Rajasthan State, India; NIST SRM 1867.

- Actinolite Asbestos: NIST SRM 1867

Adhesive - Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

Surfactant - 1 Percent aerosol OT aqueous solution or equivalent.

Isopropanol - ACS Reagent Grade.

B6.0 ANALYTICAL ELECTRON MICROSCOPY

AEM equipment requirements will not be discussed in this document; it is suggested that equipment requirements stated in the AHERA regulations be followed. Additional information may be found in the NVLAP Program Handbook for Airborne Asbestos Analysis.[3]

The following additional materials and equipment are suggested:

- Analytical balance, readable to 0.001 gram
- Ultrasonic bath
- Glass filtration assembly (25mm), including vacuum flask and water aspirator
- Mixed cellulose ester (MCE) filters (0.22μm pore size) or 0.2μm pore size polycarbonate filters
- MCE backing filters (5μm pore size)
- Silica mortar and pestle
- Beakers - glass and disposable
- Pipettes, disposable, 1,5, and 10 ml

B7.0 REFERENCES

1. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Bulk Asbestos Handbook, NISTIR 88-3879, 1988.
2. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.
3. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Program Handbook for Airborne Asbestos Analysis, NISTIR 89-4137, 1989.

APPENDIX C

Preparation and Use of Bulk Asbestos Calibration Standards

C1.0 INTRODUCTION

Evaluation of the results from national proficiency testing programs for laboratories analyzing for asbestos in bulk materials indicates that laboratories have had, and continue to have, problems with quantitation of asbestos content, especially with samples having a low asbestos concentration.[1] For such samples, the mean value of asbestos content reported by laboratories may be four to ten times the true weight percent value. It is assumed that the majority of the laboratories quantify asbestos content by visual estimation, either stereomicroscopically or microscopically; therefore, the problem of quantitation must be attributed to lack of or inadequate calibration of microscopists.

As calibration standards for asbestos-containing bulk materials are not currently commercially available, laboratories should consider generating their own calibration materials. This may be done rather easily and inexpensively.

C2.0 MATERIALS AND APPARATUS

Relatively pure samples of asbestos minerals should be obtained. Chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867) are available from NIST. A variety of matrix materials are commercially available; included are calcium carbonate, perlite, vermiculite, mineral wool/fiberglass, and cellulose. Equipment, and materials needed to prepare calibration bulk materials are listed below.

- Analytical balance, readable to 0.001 gram
- Blender/mixer; multi-speed, ~ one quart capacity
- Filtration assembly, including vacuum flask, water aspirator and/or air pump (optional)
- HEPA-filtered hood with negative pressure
- Filters, 0.4μm pore size polycarbonate (optional)
- Beakers and assorted glassware, weigh boats, petri dishes, etc.
- Hot/warm plate

- Asbestos minerals
- Matrix materials
- Distilled water.

C3.0 MATERIAL FORMULATION PROCEDURES

The formulation procedure involves first weighing appropriate quantities of asbestos and matrix material to give the desired asbestos weight percent. The following formula may be used to determine the weights of asbestos and matrix materials needed to give a desired weight percent asbestos.

$$\frac{WTa}{Wa} = \frac{WTm}{Wm}$$

Where:

WTa = weight of asbestos in grams (to 0.001 gram)
WTm = weight of matrix materials in grams (to 0.001 gram)
Wa = weight percent asbestos
Wm = weight percent matrix

Example: The desired total weight for the calibration sample is ~ 10 grams containing 5% asbestos by weight. If 0.532 grams of asbestos are first weighed out, what corresponding weight of matrix material is required?

WTa = 0.532 grams
Wa = 5%
Wm = 95%

$$\frac{0.532}{5} = \frac{WTm}{95}$$

Then: WTm = 10.108 grams

The matrix is then placed into the pitcher of a standard over-the-counter blender, the pitcher being previously filled to approximately one-fourth capacity (8-10 ounces) with distilled water. Blending is performed at the lowest speed setting for approximately ten seconds which serves to disaggregate the matrix material. The asbestos is then added, with additional blending of approximately 30 seconds, again at the lowest speed setting. Caution should be taken not to overblend the asbestos-matrix mixture. This could result in a significant reduction in the size of the asbestos fibers causing a problem with detection at normal magnification during stereomicroscopic and microscopic analyses. Ingredients of the

pitcher are then poured into a filtering apparatus, with thorough rinsing of the pitcher to ensure complete material removal. After filtering, the material is transferred to a foil dish which is placed on a hot plate. The material is covered and allowed to sit over low heat until drying is complete; intermittent stirring will speed the drying process. For fine-grained matrix materials such as gypsum, calcium carbonate, clays, etc., the sample is not filtered after the blending process. Instead, the ingredients in the pitcher are transferred into a series of shallow, glass (petri) dishes. The ingredients should be stirred well between each pouring to minimize the possible settling (and over-representation) of some components. The dishes are covered and placed on a hot plate until the contents are thoroughly dried. For small quantities of any matrix materials (15 grams or less), air-drying without prior filtering is generally very suitable for removing water from the prepared sample. For each material, the final step involves placing all formulated, dried subsamples into a plastic bag (or into one petri dish, for small quantities), where brief hand-mixing will provide additional blending and help to break up any clumps produced during drying. **All operations should be performed in a safety-hood with negative pressure.**

C4.0 ANALYSIS OF MATERIALS

All formulations should be examined with the stereomicroscope to determine homogeneity. Gravimetric analysis (ashing and/or acid dissolution) should be performed on those materials containing organic and/or acid-soluble components. Matrix materials to which no asbestos has been added should be analyzed by gravimetric analysis to determine the amount of nonashable or insoluble materials that are present. Several subsamples of each material should be analyzed by the gravimetric technique to provide information concerning the uniformity of the prepared materials. Experience has shown that the previously described formulation procedure results in relatively homogeneous materials.[2]

C4.1 Stereomicroscopic Analysis

Visual estimation of sample components using the stereomicroscope is in reality a comparison of the relative volumes of the components.[3] Therefore, differences in specific gravity between asbestos and matrix material must be considered and the relationship

between weight percent and volume percent must be determined.[4] Materials such as expanded vermiculite, perlite, and cellulose have specific gravities significantly lower than asbestos minerals. Table C1 lists the specific gravities for the three most commonly encountered asbestos varieties and several common matrix materials.

TABLE C1. SPECIFIC GRAVITIES OF ASBESTOS VARIETIES AND MATRIX MATERIALS

Asbestos Type	Specific Gravity	Matrix Type	Specific Gravity
Chrysotile	2.6	Calcium Carbonate	2.7
		Gypsum	2.3
Amosite	3.2	Perlite	~0.4
		Vermiculite (expanded)	~0.3
		Mineral Wool	~2.5
Crocidolite	3.3	Fiberglass	~2.5
		Cellulose	~0.9

The conversion of weight percent asbestos to equivalent volume percent asbestos is given by the following formula:

$$\frac{\frac{Wa}{Ga}}{\frac{Wa}{Ga} + \frac{Wm}{Gm}} \times 100 = Va$$

where:

Wa = weight percent asbestos
Ga = specific gravity of asbestos
Wm = weight percent matrix
Gm = specific gravity of matrix
Va = volume percent asbestos

Example: Chrysotile and perlite have been combined to form a 5% asbestos calibration standard, by weight. What is the equivalent volume percent asbestos?

$Wa = 5\%$
$Ga = 2.6$
$Wm = 95\%$
$Gm = 0.4$

$$Va = \frac{\frac{5}{2.6}}{\frac{5}{2.6} + \frac{95}{0.4}} \times 100 = 0.8\%$$

Conversely, to convert volume percent asbestos to equivalent weight percent, the following formula may be used.

$$\frac{(Va)(Ga)}{(Va)(Ga)+(Vm)(Gm)} \times 100 = Wa$$

Vm = volume percent matrix

Example: A calibration standard consisting of amosite and cellulose is estimated to contain 2% asbestos, by volume. What is the equivalent weight percent asbestos?

$Va = 2\%$
$Ga = 3.2$
$Vm = 98\%$
$Gm = 0.9$

$$Wa = \frac{(2)(3.2)}{(2)(3.2)+(98)(0.9)} \times 100 = 6.77\%$$

Volume percentages should be calculated for all calibration materials prepared so that visual estimates determined by examination with the stereomicroscope may be compared to true volume concentrations.

Figure C1 illustrates the relationship between volume percent and weight percent of chrysotile mixed with vermiculite and cellulose respectively. It should be noted that when asbestos in a low weight percentage is mixed with matrix materials having low specific gravities (vermiculite, perlite), the resulting volume concentration of asbestos is very low. For example, a mixture containing three percent chrysotile by weight in a cellulose matrix would result in a volume percent asbestos of approximately 1.1%; in a vermiculite matrix, the resulting volume percent asbestos would be approximately 0.4%. In the latter case especially, an analyst might possibly fail to detect the asbestos or consider it to be present in only trace amounts.

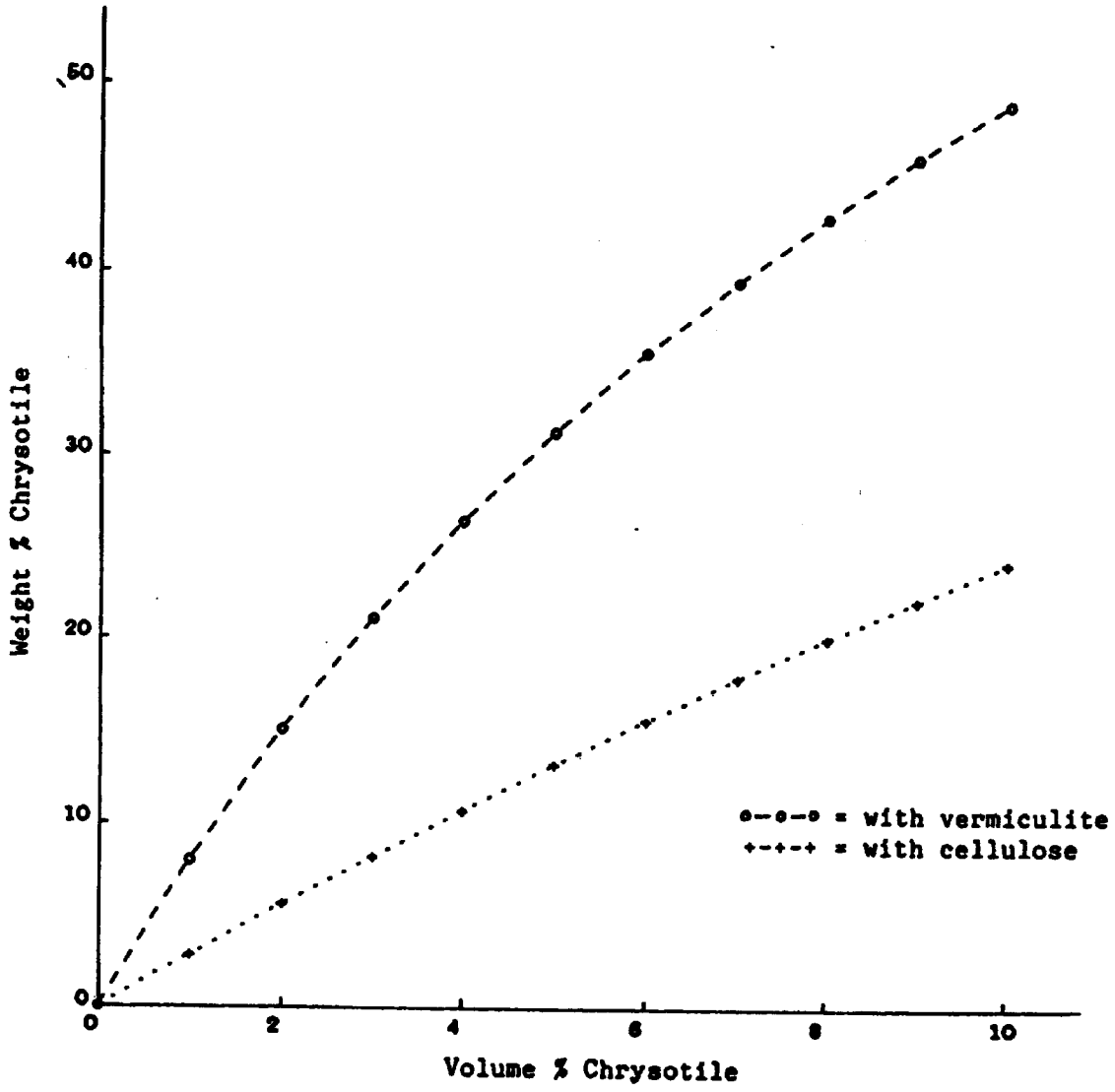


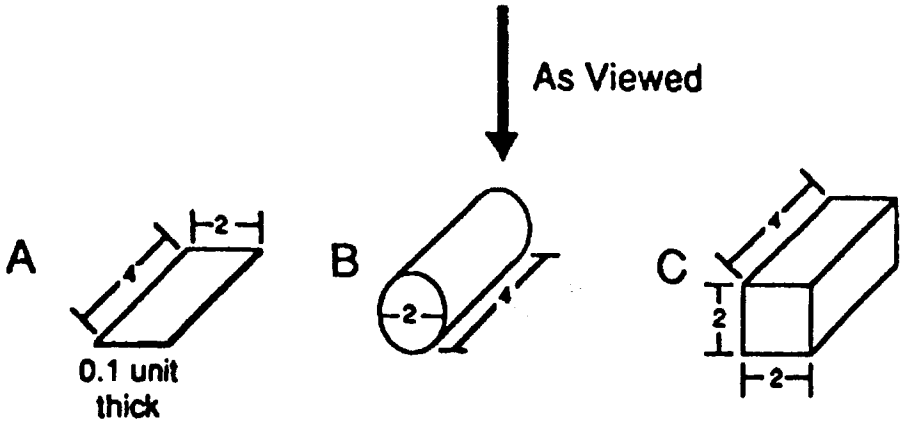
Figure C1. Relationship between volume % and weight % of chrysotile mixed with a)vermiculite and b) cellulose.

C4.2 Microscopical Analysis (PLM)

The polarized light microscope may be used to quantify asbestos and other components of a sample. Slide mounts are prepared from "pinch" samples of the calibration material and asbestos content is determined by visual area estimate and/or point counting. Both of these quantitation techniques are in fact estimates or measurements of the relative projected areas of particles as viewed in two dimensions on a microscope slide. For quantitation results to be meaningful, the following conditions should be met:

- The sample should be homogeneous for slide preparations, which are made from small pinches of the sample, to be representative of the total sample.
- Slide preparation should have an even distribution of particles and approach a one particle thickness (seldom achieved) to avoid particle overlap.
- All materials used should be identified and specific gravities determined in order to relate area percent to volume and/or weight percent.
- The size (thickness) relationship between matrix particles and asbestos fibers should be determined if the results based on projected area are to be related to volume and/or weight percent.

Particle characteristics can greatly affect the quantitation results obtained by visual area estimation or point counting. Figure C2 illustrates three hypothetical particle shapes of identical length and width (as viewed from above). Although the three-dimensional shape is different, the projected area is equal for all particles. The table accompanying Figure C2 presents data for each particle in terms of thickness, volume and projected area. It should be noted that although the projected areas may be equal, the volumes represented by the particles may vary by a factor of 20(0.8 vs 16 cubic units). It is obvious that quantitation of a sample consisting of a mixture of particles with widely ranging particle thicknesses could result in different results. For example, if a sample contained relatively thick bundles of asbestos and a fine-grained matrix such as clay or calcium carbonate, the true asbestos content (by volume) would likely be underestimated. Conversely, if a sample contained thick "books" of mica and thin bundles of asbestos, the asbestos content (by volume) would likely be overestimated.



Particle	Thickness	Volume	Projected Area
A	0.1 units	0.8 cubic units	8 sq. units
B	2 units	12.6 cubic units	8 sq. units
C	2 units	16 cubic units	8 sq. units

Note that although all particles have the same projected area, particle C volume is 20x that of particle A.

Figure C2. Relationship of projected area to volume and thickness for three different particles as viewed on a slide mount.

Table C2 illustrates several examples of expected results from area estimates or point counting of samples in which the asbestos fibers and matrix particles differ in thickness.

TABLE C2. RELATIONSHIP OF WEIGHT PERCENT, VOLUME PERCENT AND PARTICLE THICKNESS TO QUANTITATION RESULTS

Composition of Sample In Wt. %	Theoretical Vol. % Asbestos	Thickness Factor* (Matrix/Asbestos)	Expected Area %
1% Amosite 99% Calcium Carbonate	0.9	0.5	0.4
1% Amosite 99% Calcium Carbonate	0.9	1	0.9
1% Amosite 99% Calcium Carbonate	0.9	2	1.8
1% Amosite 99% Vermiculite	0.1	1	0.1
1% Amosite 99% Vermiculite	0.1	10	1.0
1% Amosite 99% Vermiculite	0.1	20	2.0
1% Amosite 99% Vermiculite	0.1	30	2.9

* Value represents the relationship between the mean thickness of the matrix particles compared to the mean thickness of the asbestos particles.

It should be noted that it is not uncommon for matrix particle thickness to differ greatly from asbestos fiber thickness, especially with matrix materials such as vermiculite and perlite; vermiculite and perlite particles may be 20 - 30 times as thick as the asbestos fibers.

The general size relationships between matrix particles and asbestos fibers may be determined by scanning slide mounts of a sample. A micrometer ocular enables the microscopist to actually measure particle sizes.

If a thickness factor can be determined for a calibration sample of known volume proportions of asbestos and matrix materials, an expected equivalent projected area asbestos can be calculated using the following formula:

$$\frac{Va}{\frac{Vm}{T} + Va} \times 100 = Aa$$

where:

Va = true volume percent asbestos
Vm = true volume percent matrix
T = thickness factor (mean size matrix particle/mean size asbestos fiber)
Aa = expected projected area percent asbestos

Example: A calibration standard of known weight percent asbestos is determined, by factoring in component specific gravities, to be 5.0% asbestos by volume. The matrix particles are estimated to be ten times thicker than the asbestos fibers. What would be the expected projected area percentage of asbestos?

Va = 5%
Vm = 95%
T = 10

$$Aa = \frac{5}{\frac{95}{10} + 5} \times 100 = 34.5\%$$

Conversely, to convert projected area percent asbestos to equivalent volume percent, the following formula may be used:

$$\frac{Aa}{T(Am) + Aa} \times 100 = Va$$

Where: Am = projected area matrix

Example: A slide containing a subsample of an amosite/mineral wool calibration standard is determined by point counting to have a projected area asbestos of 18.6%. If the mineral wool fibers are estimated to be six times the asbestos fibers, in diameter, what is the equivalent volume percent asbestos?

Am = 81.4%
Aa = 18.6%
T = 6

$$Va = \frac{(18.6)}{6(81.4) + 18.6} \times 100 = 3.67\%$$

Based on specific gravity values listed in Table 1C and on the above volume asbestos determination, what is the equivalent weight percent asbestos in the sample?

Va = 3.67%
Ga = 3.2
Vm = 96.33%
Gm = 2.5

$$Wa = \frac{(3.67)(3.2)}{(3.67)(3.2) + (96.33)(2.5)} \times 100 = 4.7\%$$

C5.0 USE OF CALIBRATION STANDARDS FOR QA/QC

Once the materials have been formulated and thoroughly characterized by all techniques to determine their suitability as calibration standards, a system for incorporating them into the QA/QC program should be established. Someone should be designated (QA officer, lab supervisor, etc.) to control the distribution of standards and to monitor the analysis results of the microscopists. Both precision and accuracy may be monitored with the use of suitable standard sets.

Records such as range charts, control charts, etc. may be maintained for volume (stereomicroscopic estimates), area (PLM) estimates and point counts. For point counts and area estimates, relatively permanent slides may be made using epoxy or Melt Mount ®. Such slides may be very accurately quantified over time as to point count values, and due to their very long shelf life, may be used for QA/QC purposes almost indefinitely.

C6.0 REFERENCES

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2. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991.

3. Perkins, R. L. and M. E. Beard, "Estimating Asbestos Content of Bulk Materials", **National Asbestos Council Journal**, Vol. 9, No. 1, 1991, pp. 27-31.

4. **Asbestos Content in Bulk Insulation Samples: Visual Estimates and Weight Composition**, U.S. Environmental Protection Agency 560/5-88-011, 1988.

APPENDIX D

Special-Case Building Materials

Asbestos laboratories are now called upon to analyze many types of bulk building materials that are very difficult to characterize by routine PLM analysis. These materials are dominantly nonfriable and can be grouped into the following categories:

- Cementitious Products (pipe, sheeting, etc.)
- Viscous Matrix Products (adhesives, cements, coatings, etc.)
- Vinyl Materials (vinyl floor tile, sheeting)
- Asphaltic Roofing Materials (shingles, roll roofing)
- Miscellaneous Products (paints, coatings, friction plates, gaskets, etc.)

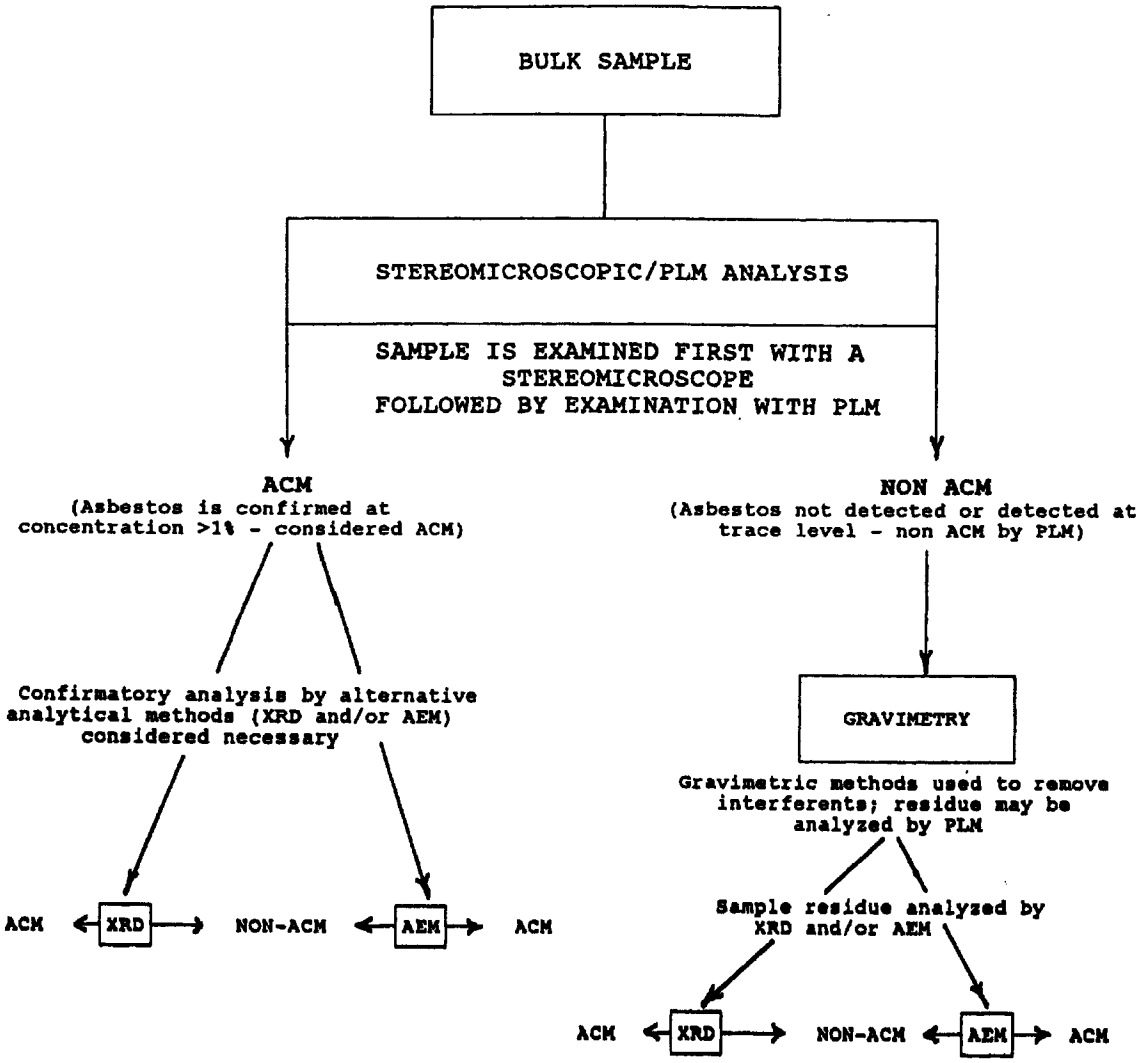
Materials characterized by interfering binder/matrix, low asbestos content, and/or small fiber size may require that additional sample treatment(s) and analysis be performed beyond routine PLM analysis. The sample treatment(s) required is(are) determined by the dominant nonasbestos sample components (see Section 2.3, Gravimetry). Materials containing an appreciable amount of calcareous material may be treated by dissolution with hydrochloric acid. Samples containing organic binders such as vinyl, plasticizers, esters, asphalts, etc. can be treated with organic solvents or ashed in a muffle furnace (preferred method) or low temperature plasma asher to remove unwanted components. Materials containing cellulose, synthetic organic fibers, textiles, etc. may also be ashed in a muffle furnace or low temperature plasma asher.

The method chosen for analysis of a sample after treatment is dependent on asbestos concentration and/or fiber size. An examination of the sample residue by PLM may disclose asbestos if the fibers are large enough to be resolved by the microscope, but additional analytical methods are required if the sample appears negative. Analysis by XRD is not fiber-size dependent, but may be limited by low concentration of asbestos and the presence of interfering mineral phases. In addition, the XRD method does not differentiate between fibrous and nonfibrous varieties of a mineral. Analysis by AEM is capable of providing positive identification of asbestos type(s) and semi-quantitation of asbestos content.

The following flowchart illustrates a possible scheme for the analysis of special-case building materials.

NOTE: Preliminary studies indicate that the XRD method is capable of detecting serpentine (chrysotile) in floor tile samples without extensive sample preparation prior to XRD analysis. XRD analysis of small, intact sections of floor tile yielded diffraction patterns that confirmed the presence of serpentine, even at concentrations of ~one percent by weight. TEM analysis of these same tiles confirmed the presence of chrysotile asbestos. With further investigation, this method may prove applicable to other types of nonfriable materials.

FLOWCHART FOR QUALITATIVE ANALYSIS OF SPECIAL CASE BUILDING MATERIALS SUCH AS FLOOR TILES, ASPHALTIC MATERIALS, VISCOUS MATRIX MATERIALS, ETC.*



*Although this flowchart is applicable to all bulk materials, it is primarily intended to be used with known problem materials that are difficult to analyze by PLM due to low asbestos concentration, and/or small fiber size, and/or interfering binder/matrix. In addition to being qualitative, the results may also be semi-quantitative. It should not be assumed that all samples need to be analyzed by AEM and XRD. The flowchart simply illustrates options for methods of analysis. Alternate methods such as SEM may be applicable to some bulk materials.

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